

CLAIMS

1. A method of evaporating liquid samples contained in at least some of a plurality of individual sample holders which are mounted within a chamber and rotated during the evaporation process so that centrifugal force is exerted on liquid contained therein during the evaporation process and wherein heat is supplied to the sample holders to heat the liquid therein whilst a pressure below atmospheric is maintained in the chamber in manner known per se, characterised in that a temperature sensing device is located in or adjacent at least one of the samples holders to sense the temperature therein at least during the evaporation process, and an electrical data signal is generated which is proportional to the sensed temperature, and the temperature data signal is conveyed via a signal path to electronic data signal processing means.
2. A method as claimed in claim 1 wherein the data signal processing means is located at the centre of rotation of the plurality of sample holders.
3. A method as claimed in claim 1 or 2 wherein the processing means converts the output of the sensor into a suitable form for transmission to an external receiver.
4. A method as claimed in claim 3 wherein the processing means converts the sensor output signals into digital signals by which a carrier signal is modulated to effect the said transmission.

5. A method as claimed in claim 3 wherein the processing means converts the sensor output signals into analogue signals by which a carrier signal is modulated to effect the said transmission.
6. A method as claimed in any of the preceding claims 3 to 5 wherein the transmitted signal constitutes a radio signal.
7. A method as claimed in claim 6 wherein the radio signals are transmitted to a receiver located externally of the housing by means of an antenna which is located externally of the housing and is connected to the signal processing means by means of a conductor which is passed through the housing wall via an insulating seal serving as a lead through.
8. A method as claimed in claim 6 or 7 wherein the chamber wall does not readily transmit, or significantly attenuates radio signals, and the radio signals from the signal processing means are received by a stationary radio receiver located within the chamber and conveyed either as radio signals or after demodulation as data signals indicative of the temperature of the sensor, via a conductive path which extends sealingly through and is insulated from the chamber wall.
9. A method as claimed in claim 8 wherein the signals are conveyed through the chamber wall as radio signals for demodulation to produce the said data signals outside the chamber.
10. A method as claimed in any of claims 3 to 5 wherein the carrier signal is a beam of light and the modulation is such as to modulate the intensity of the beam.

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11. A method as claimed in claim 10 wherein light signals are transmitted through a window which is light transmitting and which forms an integral part of the housing wall, to enable the modulated light beam to pass to a stationary light responsive device located externally of the housing and which is adapted to convert the received light signals into data signals indicative of the temperature of the sensor.
12. A method as claimed in any of claims 3 to 5 or any of claims 6 to 9 or either of claims 10 and 11 wherein the data signal is employed to drive an indicator which is calibrated to indicate sample temperature.
13. A method as claimed in any of claims 3 to 5 or any of claims 6 to 9 or any of claims 10 to 12 wherein the data signal is employed to control the source of heat heating the sample holders in the chamber.
14. A method as claimed in any of claims 1 to 13 wherein power for the processing means is derived from a battery located within a housing within which the processing means is also located.
15. A method as claimed in claim 14 wherein the battery is connected to the processing means by the closing of a motion sensitive switch which closes when the chamber rotates and is disconnected therefrom by the opening of the switch when the chamber ceases to rotate.
16. A method as claimed in any of claims 1 to 13 wherein power for the processing means is transmitted from a source external to the said housing, to a receptor located within the

housing which is connected to the processing means.

17. A method as claimed in any of claims 1 to 13 wherein power for the processing means is supplied thereto from an external power source by means of a rotational electrical connection.

18. A method as claimed in claim 17 wherein the rotational electrical connection comprises slip rings and conductive elements in contact therewith.

19. A method as claimed in claim 17 or 18 wherein the rotational electrical connection is separated from vapours in the chamber by being located outside the chamber, or inside the signal processing means housing, and seals are provided around conductors leading between the signal processing means and the external electrical connection where they pass through the wall of the chamber or the housing.

20. A method as claimed in claim 17 or 18 wherein at least one of the conductors leading between the processing means and the external rotational electrical connection, extends through the hollow interior of a drive shaft which itself extends through a seal in the chamber wall and serves to rotate both the sample holders and the said housing within the chamber.

21. A method as claimed in claim 20 wherein the drive shaft is electrically conductive and serves as one of the conductive paths for the power to the signal processing means.

22. A method as claimed in any of claims 16 to 21 wherein a material for the said housing

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is selected which is non-conductive as well as being inert in the presence of the vapours given off during the evaporation process.

23. A method as claimed in claim 22 wherein the material selected for the said housing is polypropylene.

24. A method as claimed in claim 16 or claim 22 or 23 wherein the power for the signal processing means is generated in a winding which rotates with the housing relative to a stationary magnetic flux.

25. A method as claimed in claim 24 wherein the winding is wound on soft magnetic material such as is employed to make transformer laminations.

26. A method as claimed in claim 23 or 24 wherein the magnetic flux is produced by at least one permanent magnet, which comes into close proximity with the said winding during each rotation of the sample holder, and is located either inside the chamber and the winding is in, or on, or close, to the housing, or is located outside the chamber and the winding is rotated around the internal of the chamber close to the wall thereof.

27. A method as claimed in claim 26 where the magnet is inside the chamber, and a protective coating is applied to the magnet to prevent it coming into contact with corrosive vapours in the chamber.

28. A method as claimed in any of the preceding claims wherein the sensor is sheathed in an impervious inert material so that it will not contaminate the sample or suffer

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corrosion.

29. A method as claimed in any of the preceding claims wherein the sensor is a thermocouple.

30. A method as claimed in any of the preceding claims wherein the sample holders are rotated at a speed of between 500 - 3000 rpm depending on g-force required and radius at which the samples are rotated.

31. Centrifugal evaporation apparatus comprising a vacuum chamber, a plurality of sample holders for containing separately contained liquid samples to be evaporated, which are located therein for rotation about a generally vertical axis, heating means for heating the sample holders and therefore the liquid samples therein, temperature sensitive probe means located in or adjacent at least one of the sample holders, signal path means for conveying electrical signals from the probe means to a signal processing means located within the chamber, a transmitting device also within the chamber for transmitting signals to a receiver outside the chamber, signals from the signal processing means being employed to modulate the transmitted signal so that when decoded by the remote receiver, the latter will provide a signal containing information about the temperature of the probe.

32. Apparatus as claimed in claim 31 in which the signal processing means is housed in a leak-tight housing to protect the electronic components making up the processing means from pressure fluctuations and from the vapours arising from evaporation in the chamber.

33. Apparatus as claimed in claim 31 or 32 and adapted to perform the method of claim

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12 in that it further comprises an indicating means calibrated to indicate temperature, and controlled by signals decoded by the said remote radio receiver, to indicate the temperature of the probe.

34. Apparatus as claimed in any of claims 31 to 33 adapted to perform the method of claim 13 in that it further comprises a heating means in the chamber for heating the sample holders whose heat output is controlled by the magnitude of an electrical current, and current controlling means is provided adapted to control the said electric current to the heating means, and the decoded signal from the remote radio receiver containing the temperature information is employed to control the current controlling means and thereby the heat output from the heating means and in turn the temperature to which the probe and therefore the liquid samples are permitted to rise.

35. Apparatus as claimed in any of claims 31 to 34 adapted to perform the method of claim 14 by the provision of a battery for powering the signal processing means which may be located inside or outside the housing containing the said processing means.

36. Apparatus as claimed in any of claims 31 to 34 adapted to perform the method of claim 16 by the provision of a power supply which remains stationary and is external to the housing containing the signal processing means together with a path between the power supply and the processing means for conveying power thereto as the housing rotates relative to the power supply.

37. Apparatus as claimed in any of claims 31 to 34 adapted to perform the method of claim 24 by the provision of means within the chamber but external to the housing which

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generates a stationary magnetic field, and by the provision of coil means which rotates with the housing and which is linked by the said magnetic flux and which moves relative to the flux as the housing rotates relative to the chamber, thereby to induce a current in the winding which is available to power the signal processing means, and an electrical connection is provided between the coil and the power supply circuit in the housing.

38. Methods for controlling the temperature of liquid samples in a centrifugal evaporator as described herein and with reference to and as illustrated in the accompanying drawings.

39. Apparatus for controlling the temperature of liquid samples in a centrifugal evaporator constructed and arranged to operate as described herein and with reference to and as illustrated in the accompanying drawings.

40. A method of determining the temperature of evaporating liquid samples containing or comprising at least one volatile component and contained in at least some of a plurality of individual sample holders which are mounted within a chamber and rotated during the evaporation process so that centrifugal force is exerted on volatile liquid contained therein, and wherein heat is supplied to the sample holders to heat the liquid therein whilst a pressure below atmospheric is maintained in the chamber in manner known per se, characterised in that a pressure sensing device is located in the chamber, sensing the pressure therein at least during the evaporation process, generating an electrical pressure data signal which is proportional to the sensed pressure, conveying along a signal path the pressure data signal to electronic data signal processing means which is programmed inter alia with information relating to the volatile component or components present in the samples, to convert the pressure data signal to a temperature value equal to that which

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equates to the measured vapour pressure for the known volatile component or components present.

41. A method of determining the temperature of evaporating liquid samples as claimed in claim 40, wherein the data signal processing means includes the step of addressing a look-up table containing temperature and pressure values for different liquids, and the further step of identifying to the signal processing means the volatile component or components present in the samples.

42. A method of determining the temperature of evaporating liquid samples as claimed in claim 40, wherein the data signal processing means includes an algorithm, and memory means, and the method involves the step of storing numerical values for insertion in the algorithm depending on the volatile component or components present in the samples and the pressure determined by the pressure sensing device, to enable computation of the temperature to which the sample must have been raised for the particular pressure to be observed in the chamber given the presence of the volatile component(s) concerned.

43. A method of controlling the temperature to which the samples in a centrifugal evaporator are heated by heating means within a pressure chamber containing the samples, wherein the pressure within the chamber is determined by pressure sensing means, a data signal is generated proportional to the pressure, and energy to a heating means located within the chamber for heating the samples is controlled in response to the value of the pressure signal.

44. A method of supplying heat to liquid samples in a pressure vessel in a centrifugal

evaporator, in which the samples are contained in a plurality of tubular containers which during centrifuging in the pressure vessel are swung from a generally vertical condition to a generally horizontal condition under the influence of increasing centrifugal forces, so that centrifugal forces are exerted on the liquid in the tubular containers as the pressure within the chamber is reduced, wherein radiant heat is directed towards the closed ends of the tubular containers whilst in their generally horizontal condition, thereby to achieve more uniform heating of the liquid samples.

45. A method of heating liquid samples contained in a centrifugal evaporator, wherein the samples are contained in a fixed regular array and more of the heat is directed to samples located in the central region of the array than is directed to samples around the periphery of the array.

46. Methods and apparatus as claimed in any of the preceding claims, wherein the heat source is a source of infra-red radiation.

47. Methods or apparatus as claimed in claim 46, in which a heat absorbing screen is located between the source of heat and the samples having a plurality of radiation conductive regions therein, each conductive region aligning with the position of one of the samples in the array of samples, and the thermal transmissivity of the regions increases towards the centre of the array so that samples located in the central region of the array receive more radiation per unit time than those in peripheral regions of the array.

48. Method or apparatus as claimed in any of the preceding claims, wherein the samples are contained in wells in a microtitre plate.

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50. A method of heating as claimed in any of the preceding claims, wherein the source of heating is situated at one radial position relative to the axis of rotation of the sample containers, and each sample is subjected to radiant heat energy as it passes the source of heat during its rotation around the said axis of rotation.

51. A method of heating as claimed in the preceding claims, wherein the source of heat extends around an arcuate path extending around some or all of the circular path of the samples.

52. A method of controlling the heating of liquid samples in a plurality of individual sample holders in a centrifugal evaporator wherein the samples are mounted for rotation within a vacuum chamber, the pressure of which is reduced during the evaporation process, wherein heating means is located in the chamber for heating the samples and control means is provided for controlling power to the heating means, and temperature signals from one or more probes, or a signal from a pressure sensor which detects the pressure within the chamber, are supplied to the heating control means which is programmed to provide a high heat input during early stages of the evaporation process, and smaller heat input during later stages of the process as the liquid within the samples is evaporated.

53. A method as claimed in claim 52, wherein the temperature of the samples is determined in accordance with any of the methods claimed in any of claims 40 to 42.

54. A method of controlling the heating of samples within a centrifugal evaporator wherein the samples are contained within a pressure vessel which is progressively evacuated by a vacuum pump so as to assist in the evaporation of the liquid from the samples wherein a vapour condenser is employed to increase the pumping speed to protect the vacuum pump from vapour emitted during the evaporation process, and wherein there is provided means for measuring vapour flow rate, and the method of control involves controlling the energy to the heater is controlled in response to a signal derived from the flow rate measurement such that as the flow rate decreases, the heating energy is decreased, and as the vapour flow rate approaches zero, indicating that the samples are all dry, the heat energy is shut off.

55. A method of supplying heat to a plurality of samples in each of a plurality of sample plates, each plate containing a plurality of wells or other liquid containing devices, each capable of containing one liquid sample, wherein the sample plates are supported on trays of a material having a high thermal conductivity, and the trays themselves are supported within, and have a good thermal path to and from, a support frame also formed from high thermal conductivity material, and heat is supplied to the support frame from which it is conducted by the thermal path to the trays and thereby to the samples plates and samples contained therein.

56. The method of heating a plurality of liquid samples as claimed in the preceding claim, wherein the heating is effected by infra-red radiation directed towards the said support

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frame.

57. Apparatus for supporting microtitre plates, each containing a plurality of liquid samples, for evaporation in a centrifugal evaporator, wherein heat is supplied to the microtitre plates via a supporting frame and via trays extending thereacross on which the microtitre plates are located, the trays and the frame being formed from a material having a high thermal conductivity, and wherein the region of each tray on which a microtitre plate is located, is formed with an upstanding region defining a platform adapted to fit into and engage a recessed underside of a microtitre plate located thereon, which would otherwise be spaced from the surface of the tray, thereby to improve the transfer of heat between the tray and the plate.

58. Apparatus as claimed in the preceding claim, wherein the frame and trays are formed from aluminium or copper.

59. Methods of controlling the heating of multiple samples in the pressure vessel of a centrifugal evaporator substantially as herein described and with reference to and as illustrated in the accompanying drawings.

60. Apparatus for controlling the heat to microtitre plates located within the vacuum chamber of a centrifugal evaporator constructed, arranged and adapted to operate substantially as herein described with reference to and as illustrated in the accompanying drawings.

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